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METHODS OF TEST FOR VULCANIZED RUBBERS

PART 13 TENSION SET

(First Revision)

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Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART 13 TENSION SET

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 8 September 1983, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Tension set is an important property for the rubber components subjected to tensile stress, intermittently.

0.3 The requirements for tension set in some of the present product specifications are based on different durations of time for application of strain and the formulae for calculations. The time and the formula specified in the product specifications shall be followed till they are revised to amend the value of tension set based on this test method.

0.4 This standard was originally published in 1972, and covered only three types of test pieces. In this revision, dumb-bell type test pieces are being included.

0.5 This standard is mainly based on ISO 2285-1981 'Vulcanized rubbers — Determination of tension set under constant elongation at normal and high temperatures', issued by International Organization for Standardization.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 13) prescribes the method for the determination of tension set of vulcanized natural and synthetic rubbers. This method is suitable for rubbers having the hardness within the range 30 to 94 International Rubber Hardness Degrees (IRHD), to retain their elastic properties after extension at the standard laboratory temperature, to a specified strain which is maintained for a specified time at the same or at a specified higher temperature and then released at laboratory temperature.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Tension Set — Proportion of applied strain remaining after a test piece has been stretched and allowed to retract in a specified manner.

3. OUTLINE OF THE METHOD

3.1 Test piece of standard dimension is stretched to a specified length in a suitable apparatus, for specified time and temperature and then allowed to recover. The tension set is then calculated as the percentage of the initial strain.

4. TEST PIECE

4.0 Four types of test pieces are described, strips, strips with enlarged ends, dumb-bells and rings. Tests made with different types of test pieces do not necessarily give the same value of tension set, neither shall the tests made at different temperatures necessarily place vulcanizate in the same order of merit.

4.1 Preparation

4.1.1 *Strip Test Piece* — Strips, of the material under test, between 2 and 10 mm in width and 2·0±0·2 mm thick shall be cut with a sharp die from a flat sheet. A width of 6·0 mm is preferred. The sheets may be prepared by moulding or from finished articles by cutting and buffing. Strip test pieces may have, well outside the reference length, wider ends for easier clamping. The length of any given strip depends on the selected reference length and type of straining device.

4.1.2 *Strip Test Pieces with Enlarged Ends* — Test pieces of the shape shown in Fig. 1 shall be cut with a sharp die from a flat sheet, 2·0±0·2 mm thick, of the material under test. The length of the narrow section (reference length) shall be between 25 and 50 mm. The sheets may be prepared by moulding or from finished articles by cutting and buffing.

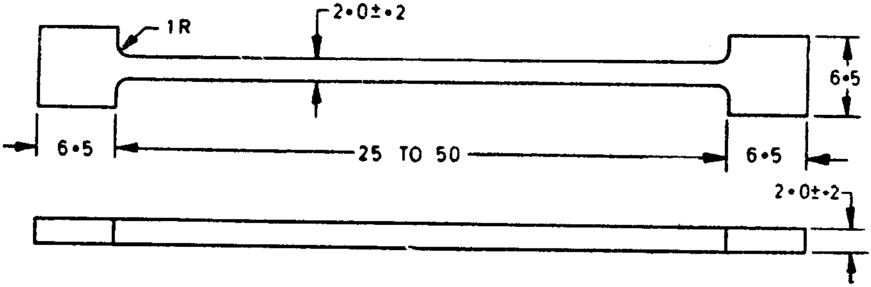

FIG. 1 TEST PIECES WITH ENLARGED ENDS

4.1.3 *Dumb-Bell Test Piece* — Dumb-bell test pieces shall have the outline shown in Fig. 2, when punched with dies, and shall conform to the dimensions given for Types 1, 2 and 3 in Table 1 when read with Fig. 1.

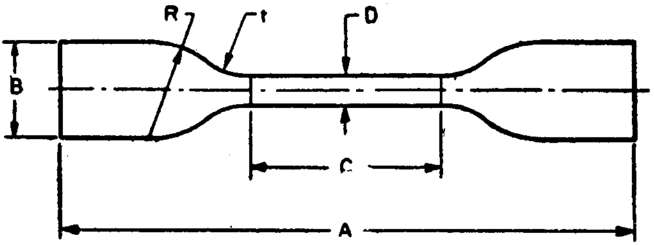

FIG. 2 DUMB-BELL TEST PIECES

4.1.3.1 Dumb-bell Type 2 and Type 3 (*see* Fig. 2 and Table 1) shall be used only in cases where there is insufficient material to enable dumb-bell, Type 1 to be used.

4.1.3.2 The bench marks shall not be more than 25 mm for Type 1, 20 mm for Type 2 and 10 mm for Type 3 and shall be equidistant from the ends of the central, parallel-sided part of the test piece.

NOTE — The use of automatic extensometers is recommended, particularly when bench marks, 10 mm apart, are used to measure the elongation.

TABLE 1 DIMENSIONS OF DUMB-BELL TEST PIECES

(*Clause* 4.1.3)

DIMENSION (1) mm	TYPE 1 (2) mm	TYPE 2 (3) mm	TYPE 3 (4) mm
Overall length (*A*), *Min*	115	75	35
Width of ends (*B*)	25±1	12·5±1·0	6±0·5
Length of narrow parallel portion (*C*)	33±2	25±1	12±0·5
Width of narrow parallel portion* (*D*)	$6^{+0\cdot4}_{-0\cdot0}$	4·0±0·1	2±0·1
Small radius (*r*)	14±1	8·0±0·5	3±0·1
Large radius (*R*)	25±2	12·5±1·0	3±0·1†
Thickness	2±0·2	2·0±0·2	1±0·1

*The variation within any one die shall not exceed 0·05 mm.

†Centre of circle on the central line of dumb-bell.

4.1.4 *Ring Test Piece* — Ring test pieces shall be cut from a flat sheet of the material under test, prepared by moulding or from finished articles by cutting and buffing, by means of a pair of concentric circular dies or rotating cutters. The separation of the two cutting edges on such dies or cutters shall not differ from the average value by more than 0·05 mm.

The following two sizes of ring test piece may be used:

Large ring test piece:

Thickness	4·0±0·2 mm
Outer diameter	52·6±0·2 mm
Inner diameter	44·6±0·2 mm

Small ring test piece:

Thickness	2·0±0·2 mm
Outer diameter	33·5±0·2 mm
Inner diameter	29·5±0·2 mm

4.2 Marking — A reference length shall be marked on the test pieces using a suitable bench marker and ink, which does not affect the material and withstands the temperature of test. The reference length for strip test pieces shall be between 25 and 50 mm. In the case of marking of ring test pieces, they shall be straightened by means of the rigid channel and the reference length, preferably 40 mm for large ring test pieces and 25 mm for small ring test pieces, marked on one of the straightened sides.

4.3 Number of Test Pieces — Three test pieces shall be used.

4.4 Time Lapse Between Manufacture and Testing

4.4.1 For all test purposes the minimum time between manufacture and testing shall be 16 hours, in order to ensure that the material attains dimensional stability due to stress relaxation.

4.4.2 In order to bind the user and supplier to a stipulated time for carrying out conformity test for supplied material, the following shall apply.

4.4.2.1 For non-product test, separate test piece is required for testing. Therefore, the maximum time between manufacture and testing shall be 8 weeks and for evaluation intended to be comparable, the tests as far as possible, should be carried out after the same time interval.

4.4.2.2 For product test, whenever possible, the time between manufacture and testing should not exceed 4 months. In other cases tests shall be made within 2 months of the date of the receipt of the product by the customer.

4.5 Conditioning — Prepared test piece shall be conditioned immediately before testing for a minimum period of 3 hours at 27±2°C, the same temperature being used throughout the test.

5. APPARATUS

5.1 Straining Device — It consists of a metal rod or other suitable guide fitted with two pairs of holders, one fixed and one movable, for the ends of the test piece. The holders shall be in the form of self-tightening clamps for strip test pieces, in the form of jaws to hold tap ends in a firm position for strip test pieces with enlarged ends and in the form of flat pulleys of about 5 mm width and 10 mm diameter for ring test pieces. If so desired, means of operating the moving holder other than by hand may be provided, for example, a screwed rod, provided that the tolerances on extension speed are met (*see* **6.1**). Suitable stops or graduations may also be provided, to avoid over-extension in the initial straining of the test piece.

Straining devices shall be so designed that when used at high temperature in an oven, they can be placed with the reference lengths of the test pieces perpendicular to the direction of the air flow; they shall also be of minimal mass in order to avoid excessive lag in attainment of temperature equilibrium after introduction into an oven.

Multiple unit straining devices may be used, provided the foregoing requirements are met.

5.2 Measuring Device — Suitable for measuring length to the nearest 0·1 mm.

5.2.1 For strip test pieces a bench marker shall be provided to mark the length used for measurement, hereinafter called the reference length.

5.2.2 For strip test pieces with enlarged ends the length of the narrow portions shall be used as the reference length.

5.2.3 For ring test pieces the reference length may alternatively be the inner diameter of the ring, in which case a graduated cone, allowing measurements to be made to the nearest 0·1 mm, may be used. If measurements are made on a straight reference length, a rigid channel, 3·5 mm deep and 20 mm wide for large ring test pieces, and 1·75 mm deep and 10 mm wide for small ring test pieces, shall be provided and for strengthening portions of such test pieces during marking and measuring of the reference length.

6. PROCEDURE

6.1 Measure the unstrained reference length to the nearest 0·1 mm at 27±2°C. Place the ends of strip test pieces into the clamps, or tab ends of test pieces with enlarged ends into the jaws, or fitring test pieces over the pulley rims of the straining device. Extend the test pieces to the required strain at a speed between 2 and 10 mm/s.

With ring test pieces, rotate the pulleys slightly by hand to equalize the strain in the two halves of the ring, ensuring that the reference length remains centralized between the pulleys.

6.2 Between 10 and 20 minutes after the specified strain has been reached, measure the strained reference length to the nearest 0·1 mm. When using the inner diameter of ring test pieces as the reference length, it may be calculated from the diameter of the pulleys and the distance between them, measured to the nearest 0·1 mm. If the strain, calculated as described in **7.1**, does not conform to the appropriate standard value after taking account of tolerances, discard the test pieces, prepare a fresh test piece with a modified applied strain.

6.3 Tests at Elevated Temperatures

6.3.1 For tests at other than standard laboratory temperatures, place the strained test pieces into the oven operating at the test temperature: Between 20 and 30 minutes after the specified strain has been reached; at the end of the test period, remove the test pieces from the oven and allow to cool in the strained condition for 30 to 35 minutes.

6.3.2 Release the strain at a speed of 2 to 10 mm/s, remove the test pieces from the clamps or pulleys and lay free on a wooden surface. After 30^{+3}_{-0} minutes measure the reference length to the nearest 1·0 mm, using the rigid channel to straighten ring test pieces, if required.

6.3.3 The common elevated temperatures at which this test may be carried out are 70±1°C, 85±1°C, 100±1°C, 125±2°C and 150±2°C. If a temperature other than standard laboratory temperature is used, 70±1°C shall be preferred. For special cases, higher standard temperature may be used.

7. STRAIN AND DURATION OF TEST

7.1 Strain

7.1.1 *Calculation*

$$\text{Percentage strain} = 100 \frac{l_s - l_0}{l_0}$$

where

l_0 = original unstrained reference length, and
l_s = strained reference length, which shall be one of the following values:

percent
(25±2·5)
(50±5)
(100±10)
(200±10)
(300±10)

7.1.2 The strain value should be selected in accordance with the final application of the vulcanizate and with reference to its breaking elongation and the test temperature. The value (100±10) percent is preferred unless the above considerations dictate otherwise.

7.2 Duration of Test — The test pieces shall be strained for 24^{+2}_{-0} hours or alternatively, if a longer test period is required, 72^{+2}_{-0} hours or 168^{+2}_{-0} hours, the test period commencing 30 minutes after the original straining of the test pieces.

8. CALCULATION

8.1 Calculate the tension set as a percentage of initial strain:

$$\text{Tension set} = 100 \frac{l_1 - l_0}{l_s - l_0}$$

where

l_1 = reference length after recovery,
l_o = unstrained reference length, and
l_s = strained reference length.

8.1.1 The mean of the results for the three test pieces shall be reported, if the individual value for the three test results agree within 10 percent of the mean. If they do not, the test shall be repeated using three further test pieces and the same procedure used for them. If they do not agree with 10 percent of the mean, median of six test results shall be reported.

9. TEST REPORT

9.1 The test report shall include the following information:

a) Method of preparation of the test pieces, for example, whether moulded or cut;
b) A full description of the sample and any relevant facts about its pre-test history;
c) Curing condition applied to the test pieces, if known;
d) Duration and temperature of conditioning of the test pieces prior to testing;
e) Type and dimensions of the test pieces;
f) Number of test pieces tested;
g) Strain applied;
h) Duration and temperature of test;
j) Any non-standardized procedure adopted;
k) Mean or median value of the tension set, in percent, for test pieces tested; and
m) Date of the test.

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